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IS 1448-41 (1992): Methods of test for petroleum and its products, Part 41: Water and sediment in crude oils and fuel oils by centrifuge [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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IS 1448 [P : 41] : 1992
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पेट्रोलियम और इसके उत्पादों की परीक्षण पद्धतियाँ

[पी : 41]

अपकेन्द्रण द्वारा कच्चे तेलों और ईंधन तेलों में पानी और तलछट

(तीसरा पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 41]

WATER AND SEDIMENT IN CRUDE OILS AND FUEL OILS BY CENTRIFUGE

(Third Revision)

First Reprint AUGUST 1997

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 1992 **Price Group 4**

Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee, PCD 1

FOREWORD

This Indian Standard [P : 41] was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Test for Petroleum, Petroleum Products and Lubricants Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 41]

WATER AND SEDIMENT IN CRUDE OILS AND FUEL OILS BY CENTRIFUGE

(Third Revision)

Adapted from joint publication ASTM Designation D 1796 : 83/IP Designation 75/82/API Designation : Manual of Petroleum Measurement Standards Chapter 10 : 6 (MPMS) and ASTM D 4007-81/IP 359/82/API Designation Chapter 10.3 (MPMS)

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1 SCOPE

1.1 This standard covers the laboratory determination of water and sediment in crude oils and fuels oils by means of the centrifuge procedure.

1.2 This centrifuge method for determining water and sediment in crude oils is not entirely satisfactory. The amount of water detected is almost always lower than the actual water content. When a highly accurate value is required, the revised procedures for water by distillation { IS 1448 [P : 40] : 1987 'Methods of test for petroleum and its products : Part 40 Water by distillation (*third revision*)' } and sediment by extraction { IS 1448 [P : 30] : 1970 'Methods of test for petroleum and its products : Part 30 Sediment in crude and fuel oils by extraction (*first revision*)'} must be used.

1.3 With some types of fuel oils it is difficult to obtain water or sediment contents with this method.

1.4 This standard may involve hazardous materials, operations and equipments. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

NOTE — The Committee felt that data should be generated by various refineries regarding the applicability of this method for the range of API gravity for inclusion in the standard at a later date.

SECTION 1 DETERMINATION OF WATER AND SEDIMENT IN CRUDE OILS

2 SUMMARY OF METHOD

2.1 Equal volumes of crude oil and water saturated toluene are placed into a cone-shaped centrifuge tube. After centrifugation, the volume of the higher gravity water and sediment layer at the bottom of the tube is read.

3 SIGNIFICANCE AND USE

3.1 The water and sediment content of crude oil is significant because it can cause corrosion of equipment and problems in processing. A determination of water and sediment content is required to measure accurately net volumes of actual oil in sales, taxation, exchanges, and custody transfers.

4 APPARATUS

4.1 Centrifuge

4.1.1 A centrifuge capable of spinning two or more filled cone-shaped, 203 mm (8 in) centrifuge tubes at a speed which can be controlled to give a relative centrifugal force (rcf) of a minimum of 600 at the tip of the tubes shall be used.

4.1.2 The revolving head, trunnion rings, and trunnion cups, including the cushions shall (Caution) be soundly constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall firmly support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to eliminate danger if any breakage occurs.

4.1.3 The centrifuge shall be heated and (Caution) should be controlled thermostatistically to avoid unsafe conditions. It should be capable of maintaining the sample temperature during the entire run at 60° ± 3°C (140° ± 5° F).

4.1.4 *Caution*

Electric powered and heated centrifuges must meet all safety requirements for use in hazardous areas.

4.1.5 Calculate the speed of the rotating head in revolutions per minute (rpm) as follows:

$$\text{rpm} = 1\,335 \sqrt{rcf/d}$$

where

rcf = relative centrifugal force

d = diameter of swing (mm) measured between tips of opposite tubes when in rotating position.

or,

$$\text{rpm} = 265 \sqrt{rcf/d}$$

where

rcf = relative centrifugal force, and

d = diameter of swing (mm) measured between tips of opposite tubes when in rotating position.

4.2 Centrifuge Tubes

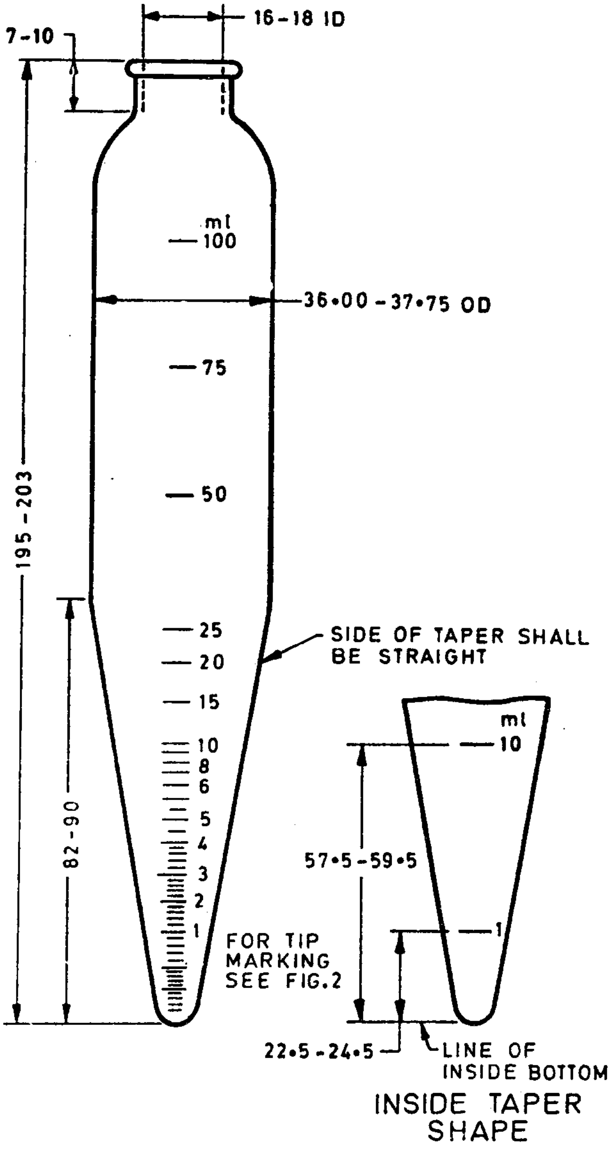
4.2.1 Each centrifuge tube shall be a 203 mm (8 in) cone-shaped tube, conforming to dimensions given in Fig. 1 and made of thoroughly annealed glass. The graduations, numbered as shown in Fig. 1 shall be clear and distinct, and the mouth shall be constricted in shape for closure with a cork. Scale error tolerance and the smallest graduations between various calibration marks are given in Table 1 and apply to calibrations made with air-free water at 20° C (68° F), when reading the bottom of the shaded meniscus.

Table 1 Centrifuge Tube Calibration Tolerance for 8-in (203 mm) Tube

Range (ml)	Sub-division (ml)	Volume Tolerance (ml)
0 to 0·1	0·05	± 0·02
Above 0·1 to 0·3	0·05	± 0·03
Above 0·3 to 0·5	0·05	± 0·05
Above 0·5 to 1·0	0·10	± 0·05
Above 1·0 to 2·0	0·10	± 0·10
Above 2·0 to 3·0	0·20	± 0·10
Above 3·0 to 5·0	0·50	± 0·20
Above 5·0 to 10·0	1·0	± 0·50
Above 10·0 to 25·0	5·0	± 1·00
Above 25·0 to 100·0	25·0	± 1·00

4.3 Bath

4.3.1 The bath shall be either a solid metal block bath or a liquid bath of sufficient depth for immersing the centrifuge tube in the vertical position to the 100 ml mark. Means shall be provided for maintaining the temperature at 60 ± 3°C (140 ± 5° F) (*see* Note under **5.1.1**).



NOTE — For volumetric tolerances, *see* Table 1.

All dimensions in millimetres.

FIG. 1 CENTRIFUGE TUBE

5 REAGENES

5.1 Toluene — *See* IS 1839 : 1961 'Toluene, reagent grade.

5.1.1 Typical specifications for this material are:

$C_6H_5CH_3$	FW 92·14
Colour (APHA)	10
*Boiling range (initial to dry point)	2·0°C
Residue after evaporation	0·001 percent
Substances darkened by H_2SO_4	passes A.C.S. test
Sulphur compounds (as S)	0·003 percent
*Recorded boiling point 110·6°C.	

The solvent shall be water saturated at 60 ± 3°C (140 ± 5°F) but shall be free of suspended water. See Annex A for the solvent water saturation procedure.

NOTE — By contractual agreement 49 ± 3° (120 ± 5°F) may be used.

5.2 Demulsifier

5.2.1 A demulsifier should be used to promote the separation of water from the sample and to prevent its slinging to the walls of the centrifuge tube. The recommended stock solution is 25 percent demulsifier to 75 percent toluene. For some crude oils a different ratio of demulsifier to toluene may be required. Demulsifiers used in the concentration and quantity recommended will not add to the water and sediment volume determined. The solution must be stored in a dark bottle that is tightly closed.

NOTE — Typical demulsifier like PETROLITE DS-655 (Petrolite. USA); QF-319 (Quick Chemicals, USA) TK-343 (Toho India Cactus, Japan), NALCO 5541 (NALCO India, Calcutta), HICO D 2200 (HICO, Bombay) or equivalent may be used.

6 PRECAUTIONS

6.1 Toluene Warning. Flammable. Keep away from heat, spark and open flame. Vapour harmful. Toluene is toxic. Particular care must be taken to avoid breathing the vapour and to protect the eyes. Keep container closed. Use with adequate ventilation. Avoid prolonged or repeated contact with the skin. In case of a spill, soak up with sand or diatomaceous earth. In case of fire, use water spray, foam, dry chemical, or carbon dioxide.

6.2 Centrifuge Warning. Note all Cautions of **4.1**.

7 SAMPLING

7.1 Sampling is defined as all steps required to obtain an aliquot of the contents of any pipe, tank, or other system and to place the sample into the laboratory test container.

8 PROCEDURE

8.1 Fill each of two centrifuge tubes (**4.2**) to the 50 ml mark with sample directly from the sample container. Then, with a pipette, add 50 ml of toluene, which has been water saturated at 60°C (140°F) or 49°C (120°F) (*see* Note under **5**). Read the top of the meniscus at both the 50 ml and 100 ml marks. Add 0·2 ml of demulsifier solution (**5.2**) to each tube, using a 0·2 ml pipette. An automatic pipettor may be used. Stopper the tube tightly and invert the tubes 10 times to ensure that the oil and solvent are uniformly mixed.

8.2 In the case where the crude oil is very viscous and mixing of the solvent with the oil would be difficult, the solvent may be added to the centrifuge tube first to facilitate mixing. Care must be taken in order not to fill the centrifuge tube past the 100 ml mark with the sample.

8.3 Loosen the stoppers slightly and immerse the tubes to the 100 ml mark for at least 15 min in the bath maintained at 60 ± 3°C (140°F) (*see* Note 1). Secure the stoppers and again invert the tubes 10 times to ensure uniform mixing of oil and solvent. [Caution : The vapour pressure at 60°C (140°F) is approximately double that at 40°C (104°F)].

8.4 Place the tubes in the trunnion cups on opposite sides of centrifuge to establish a balanced condition (*see* Note 2) . Retighten the corks and spin for 10 min at a minimum relative centrifugal force of 600, calculated from the equation given in **4.1.5**.

8.5 Immediately after the centrifuge comes to rest following the spin, read and record the combined volume of water and sediment at the bottom of each tube to the nearest 0·05 ml from 0·1 ml to 1 ml graduations and to the nearest 0·1 ml above 1 ml graduations. Below 0·1 ml, estimate to the nearest 0·025 ml (refer to Fig. 2). Return the tubes without agitation to the centrifuge and spin for another 10 min at the same rate.

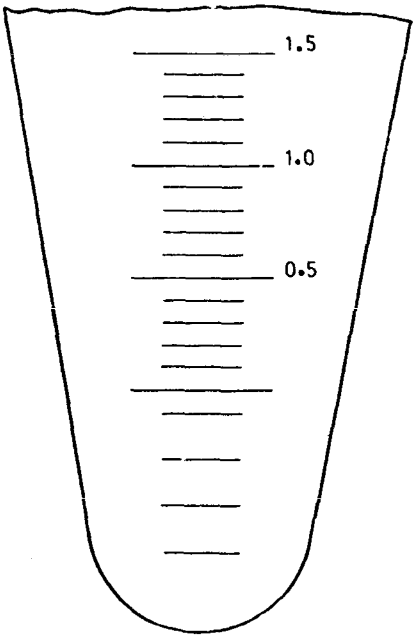


FIG. 2 TIP OF CENTRIFUGE TUBE

8.6 Repeat this operation until the combined volume of water and sediment remains constant for two consecutive readings. In general, not more than two spinnings are required.

NOTES

1 The temperature of the sample during the entire centrifuge procedure should be maintained at 60 ± 3°C (140° ± 5°F) (*see* Note under **5**).

2 To avoid the danger of tubes breaking in the cups, care must be taken that the tubes are bedded onto the bottom cushion so that no part of the tube is in contact with the rim of the cup.

9 CALCULATION

9.1 Record the final volume of water and sediment in each tube. If the difference between the two readings is greater than one subdivision on the centrifuge tube (*see* Table 1) or 0·025 ml for readings of 0·10 ml and below, the readings are inadmissible and the determination shall be repeated.

9.2 Express the sum of the two admissible readings as the percentage by volume of water and sediment; report in the results as shown in Table 2.

10 PRECISION AND ACCURACY

10.1 The precision of this method, as obtained by statistical examination of interlaboratory test results in the range of 0·01 to 1·0 percent, is described in **10.1.1** and **10.1.2**.

Table 2 Expression of Results

(Readings in ml)

Tube 1	**Tube 2**	**Total Percent Water and Sediment**
No visible water and sediment	No visible water and sediment	—
No visible water and sediment	0·025	0·025
0·025	0·025	0·05
0·025	0·05	0·075
0·05	0·05	0·10
0·05	0·075	0·125
0·075	0·075	0·15
0·075	0·10	0·175
0·10	0·10	0·20
0·10	0·15	0·25

NOTE — For volumetric tolerance, *see* Table 1.

10.1.1 *Repeatability*

The difference between successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty:

> From 0·0 percent to 0·3 percent water, *see* Fig 3.
>
> From 0·3 percent to 1·0 percent water, repeatability is constant at 0·12.

10.1.2 *Reproducibility*

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the following value in only one case in twenty:

> From 0·0 percent to 0·3 percent water, *see* Fig. 3.
>
> From 0·3 percent to 1·0 percent water, reproducibility is constant at 0·28.

SECTION II DETERMINATION OF WATER AND SEDIMENT IN FUEL OILS

11 SUMMARY OF METHOD

11.1 Equal volumes of fuel oil and water saturated toluene are placed in cone-shaped centrifuge tube. After centrifugation, the volume of the higher gravity water and sediment layer at the bottom of the tube is read.

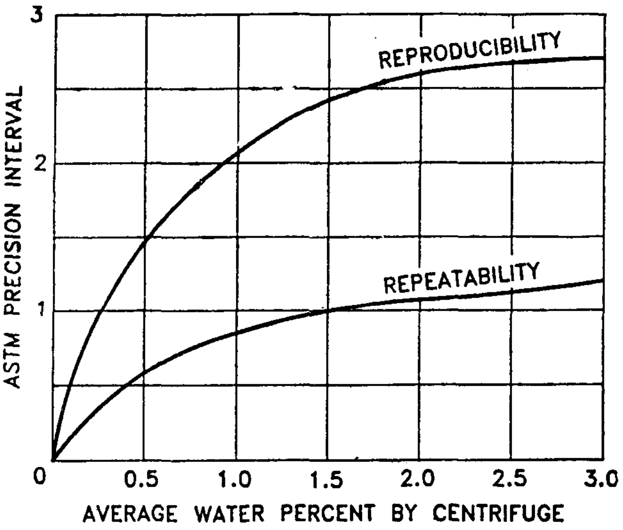


FIG. 3 BASIC SEDIMENT AND WATER PRECISION

12 SIGNIFICANCE

12.1 The water and sediment content of fuel oils is significant because it can cause corrosion of equipment and problems in processing. A determination of water and sediment content is required to measure accurately net volumes of actual fuel oil in sales, taxation, exchanges and custody transfers.

13 SOLVENT

13.1 Toluene — *See* **5.1**.

13.2 The solvent shall be water saturated at ambient temperature, but shall be free of suspended water. This may be accomplished by the additional of 2 ml of water per 1 000 ml of solvent. Shaking will aid in saturation, but adequate settling time is necessary to ensure that the solvent is free of suspended water before use.

NOTE — Some oils may require other solvents or solvent-demulsifier combinations. Those agreed upon between the purchaser and the seller may be used.

14 APPARATUS

14.1 Centrifuge

14.1.1 A centrifuge capable of spinning two or more filled cone-shaped 203-mm (8-in) centrifuge tubes at a speed which can be controlled to give a relative centrifugal force (*rcf*) of between 500 and 800 at the tip of the tubes shall be used.

14.1.2 The revolving head, trunnion rings, and trunnion cups, including the cushions, shall be soundly constructed to withstand the maximum centrifugal force capable of being delivered by the power source. The trunnion cups and cushions shall firmly support the tubes when the centrifuge is in motion. The centrifuge shall be enclosed by a metal shield or case strong enough to eliminate danger if any breakage occurs.

14.1.3 Calculate the speed of the rotating head in revolutions per minute (rpm) as follows:

$$\text{rmp} = 1\,355\sqrt{rcf/d}$$

where

rcf = relative centrifugal force, and

d = diameter of swing, mm measured between tips of opposite tubes when in rotating position.

or,

$$\text{rpm} = 265\sqrt{rcf/d}$$

where

rcf = relative centrifugal force, and

d = diameter of swing (inches) measured between tips of opposite tubes when in rotating position.

14.2 Centrifuge Tubes

14.2.1 Each centrifuge tube shall be a 203-mm (8-in) cone-shaped tube, conforming to the dimensions given in Fig. 1 and made of thoroughly annealed glass. The graduations, numbered as shown in Fig. 1, shall be clear and distinct, and the mouth shall be constricted in shape for closure with a cork. Scale error tolerances and the smallest graduations between various calibration marks are given in Table 1 and apply to calibrations made with air-free water at 20°C (68° F), when reading the bottom of the shaded meniscus.

14.3 Bath

14.3.1 The bath shall be either a solid metal block bath or a liquid bath of sufficient depth for immersion the centrifuge tube in the vertical position to the 100 ml mark. Means shall be provided for maintaining the temperature at 49 ± 1°C (120° ± 2°F) and 60 ± 1°C (144° ± 2°F).

15 SAMPLING

15.1 Sampling is defined as all steps required to obtain an aliquot of the contents of any pipe, tank, or other system and to place them into the laboratory test container.

16 PROCEDURE

16.1 Fill each of two centrifuge tubes (**14.2**) to the 50-ml mark with solvent; than immediately pour the well-shaken sample directly from the sample container into the centrifuge tubes until the total volume in each tube is 100 ml. Read the top of the meniscus, at both the 50 and 100-ml marks. Stopper the tubes tightly and shake vigorously until the contents are thoroughly mixed. Immerse the tubes to the 100-ml mark for 10 min in the bath maintained at 49 ± 1°C (120 ± 2°F).

NOTE — If wax contributes to the volume of water and sediment observed, preheat the oil solvent mixture to 60°C (140°F) before each whirling; the final temperature of the mixture shall not drop below 46°C (115°F).

16.2 Invert the tubes to assure that the oil and solvent are uniformly mixed and shake cautiously. (Caution — The vapour pressure at 60°C (140°F) is approximately double that at 38°C (100°F).

16.2.1 Place the tubes in the trunnion cups on opposite sides of the centrifuge to establish a balanced condition, and spin for 10 minutes at a rate calculated from the equation given in **14.1**, sufficient to produce a relative centrifugal force (rcf) of between 500 and 800 at the tip of the whirling tubes (*see* Table 3 for the relationship between diameter of wing, relative centrifugal force, and revolutions per minute).

16.2.2 Immediately after the centrifuge comes to rest following the spin, read and record the combined volume of water and sediment (*see* Note under **16.1**) at the bottom of each tube to the nearest 0·05 ml from 0·1 to 1 ml graduations and to the nearest 0·1 ml above the 1-ml graduations. Below 0·1 ml estimate to the nearest 0·025 ml (*see* Fig. 2). Return the tubes without agitation to the centrifuge and spin for another 10 minutes at the same rate.

16.2.3 Repeat this operation, until the combined volume of water and sediment remains constant for two consecutive readings. In general, not more than two spinnings are required.

17 CALCULATION

17.1 Record the final volume of water and sediment in each tube and report the sum of these two readings as the percentage of sediment and water. Report results lower than 0·05 percent either as 0 or 0·05, whichever is closer.

17.2 Report the solvent used, if other than toluene. Report the name and amount of demulsifier, if used. Report the bath temperature.

Table 3 Rotation Speeds Applicable for Centrifuge of Various Diameters of Swing

Diameter of Swing (in)	Revolution per Minute at 500 *rcf*	Revolution per Minute at 800 *rcf*
12	1 710	2 160
13	1 650	2 080
14	1 590	2 000
15	1 530	1 930
16	1 480	1 870
17	1 440	1 820
18	1 400	1 770
19	1 360	1 720
20	1 330	1 680
21	1 300	1 640
22	1 270	1 600
23	1 240	1 560
24	1 210	1 530

*Measured in inches between tips opposite tube when in rotating position.

18 PRECISION

18.1 The criteria described in **18.1.1** and **18.1.2** should be used for judging the acceptability of results (95 percent probability).

18.1.1 *Repeatability*

The difference between successive test results under constant operating conditions on identical test material, would, in the long run, in the normal and correct operation of the test method, exceed the values in Fig. 4 in only one case in twenty.

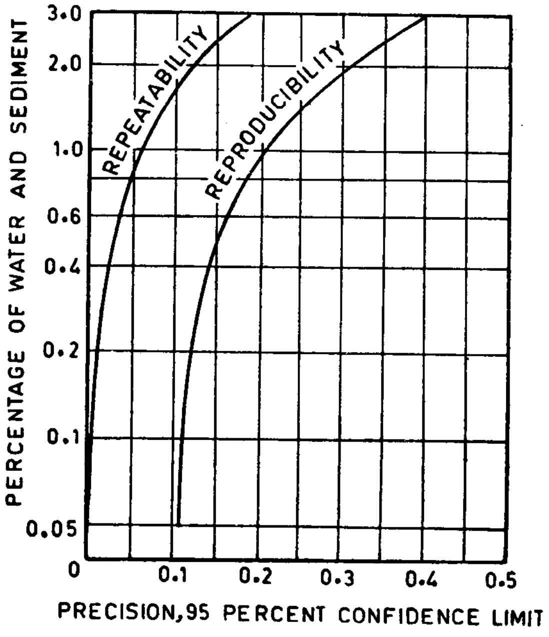


FIG. 4 PRECISION CURVES FOR CENTRIFUGE TUBE METHODS

18.1.2 *Reproducibility*

The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material, would, in long run, in normal and correct operation of the test method, exceed the values in Fig. 4 in only one case in twenty.

ANNEX A

(*Clause* 5.1.1)

PROCEDURE TO WATER SATURATE TOLUENE

A-1 GENERAL

A-1.1 This method is satisfactory for the water saturation of toluene to be used for determination of water and sediment in crude oil by centrifuge method.

A-2 SIGNIFICANCE

A-2.1 Figure 5 shows that water is soluble in toluene to a significant extent. The percentage of water that will dissolve increases as the temperature is increased from about 0·03 percent at 21°C (70°F) to about 0·17 percent at 70°C (158°F). Toluene, as normally supplied, is relatively dry and if used in an as-received condition, will dissolve a portion of or even all of any water present in a crude oil sample. This would reduce the apparent sediment and water level in the crude sample. To determine water and sediment accurately by centrifuge on a crude oil sample, the toluene must first be saturated at the centrifuge test temperature.

A-3 REAGENTS

A-3.1 Toluene — *See* **5.1**.

A-3.2 Water — Either distilled or tap water, shall be used (*see* IS 1070 : 1977).

A-4 APPARATUS

A-4.1 A liquid bath of sufficient depth for immersing a 1-quart or 1-litre bottle to its shoulder shall be used. Means shall be provided for maintaining the temperature at 60 ± 3°C (140 ± 5°F).

A-4.2 A 1-quart or 1-litre glass bottle with screw top shall be used.

A-5 PROCEDURE

A-5.1 Adjust the heating bath to the temperature at which the centrifuge test is to be run. Maintain the bath temperature to ± 3°C.

A-5.2 Fill the glass bottle with 700-800 ml of toluene. Add 25 ml of water. Screw the cap on the bottle and shake vigorously for 30 s.

A-5.3 Loosen the cap and place the bottle in the bath for 30 min. Remove the bottle, tighten the cap, and shake cautiously for 30 s.

A-5.4 Repeat above pressure (**A-5.3**) three times.

CAUTION : The vapour pressure toluene at 60°C (140°F) is approximately twice that at 38°C (100°F).

A-5.4.1 Allow the bottle with the water-toluene mixture to sit in the bath 48 h before using. This will ensure complete equilibrium between the toluene and the free water as well as complete saturation at the desired temperature. If it is necessary to use the water-saturated toluene before the 48 h equilibration time has been completed, the solvent must be poured into centrifuge tubes and centrifuged in the same equipment at the same relative centrifugal force and temperature that is used for the centrifuge test. The toluene must be carefully pipetted from the centrifuge tube so that any free water that may be in the bottom of the tube is not disturbed.

A-5.4.2 Saturation is time and temperature dependent. It is recommended that bottles of the toluene-water mixture be kept at test temperature in the bath at times so that saturated solvent will be available whenever tests are to be run.

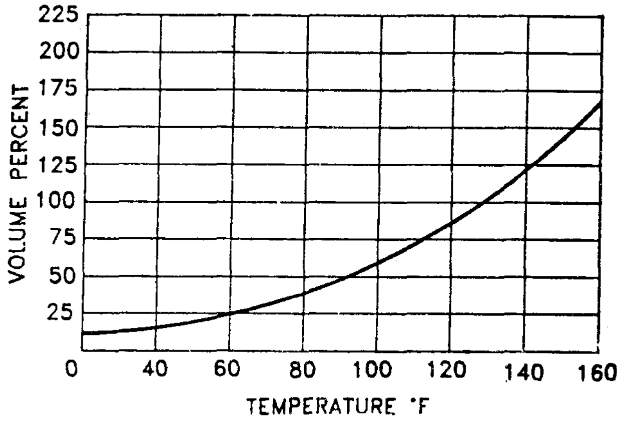


FIG. 5 SOLUBILITY OF WATER IN TOLUENE

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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards Monthly Additions'.

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